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CERTIFICATE

This certificate is issued in support of an application for Patent registration in a country outside New Zealand pursuant to the Patents Act 1953 and the Regulations thereunder.

I hereby certify that annexed is a true copy of the Provisional Specification as filed on 18 February 2004 with an application for Letters Patent number 531217 made by NEW ZEALAND FOREST RESEARCH INSTITUTE LIMITED.

Dated 3 March 2005.

Neville Harris

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Commissioner of Patents, Trade Marks and Designs

COMMISSIONER OF PATENTS, TRADEMARKS & DESIGNS
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531217

NEW ZEALAND

PATENTS ACT, 1953

PROVISIONAL SPECIFICATION

IMPREGNATION PROCESS

We, NEW ZEALAND FOREST RESEARCH INSTITUTE LTD, a New Zealand company of Sala Street, Rotorua, New Zealand, do hereby declare this invention to be described in the following statement:

IMPREGNATION PROCESS

FIELD OF THE INVENTION

This invention relates generally to impregnation processes for impregnating wood or wood products to reduce improve the decay resistance, dimensional stability and UV resistance of the wood and densify the wood and in particular but not exclusively to an acetylation impregnation process.

BACKGROUND

Wood and wood based products have some undesirable properties that need to be addressed in order to improve the performance characteristics of wood and to extend their application to new fields. There are a number of the undesirable properties that can be improved by modification, for example dimensional instability due to the hygroscopic nature of wood, discoloration, biological degradation and degradation on exposure to UV light.

Many of the conventional leading technologies for treating wood or wood based materials comprise impregnation with heavy metal preservatives. However, the utilization of heavy metals in preservatives for timber treatment is not now favoured due to toxicity and environmental issues.

Acetylation of solid wood has been extensively investigated in the past, but due to the lack of an appropriate processing system to impregnate, react and recover the by-product of impregnation in a time period to be feasible, its commercial application has been limited. However acetylation is environmentally friendly and can improve all or some of the aforementioned shortcomings of wood.

Acetylation of the wood prevents water molecules from penetrating the cell wall by bulking the cell wall and substituting the hygroscopic hydroxyl groups of lignin and hemicellulose with hydrophobic (less polar) acetyl groups. Acetylation treatment for dimensional stabilization and biological resistance has been extensively studied. In addition the impact of acetylation on weathering effects, discoloration and degradation by exposure to UV light has been examined.

Acetylation of solid wood is traditionally practiced in several steps. Typically, impregnation is first performed in a pressure cylinder and the acetylation reaction is then performed in a separate dedicated plant to heat and reflux the acetic anhydride for a lengthy period until acetylation is complete. After treatment and heat reaction, the by-product, which is a mixture of unreacted acetic anhydride and acetic acid, is recovered. European patent 0 213 252 discloses a process in which wood was "impregnated by acetic anhydride using a vacuum or vacuum pressure technique, the material was then drained of excess acetic anhydride and placed in a chamber heated to 120° C. The material was heated at this temperature for 2 to 8 hours". European patent EP 0 680 810 A1 and B1 discloses a process in which wood was impregnated by acetic anhydride at a temperature in the range of 80 – 150°C preferably 90 – 130°C and then maintained at a temperature between 70-150°C for a period of time up to 24 hours (suitably about 3 hours) to cause the acetylation reaction within the wood.

The effect of different catalysts on the reaction rate has also been investigated, but heating to temperatures of up to 139°C, which is the boiling point of acetic anhydride, has proved to be the best option. Some researchers have attempted to use gaseous acetylation but gaseous phase treatment of solid wood achieves insufficient loading or weight gain for improvement in stability and decay resistance. To achieve a 15% weight gain in acetylation theoretically 30% or more acetic anhydride is required. To impregnate gaseously this amount of acetic anhydride in solid wood requires an extremely long impregnation time.

Gaseous acetylation of wood at high temperature has only been successful on very small wood particles such as fibers, flakes, chips or sawdust. United States patent 6,376,582 discloses use of vapour of the acetylating agent at a reaction temperature of around 140 to about 210°C at atmospheric pressure. Thus the gaseous acetylation of solid wood is restricted to very small wood dimensions as the rate of vapour-phase acetylation is determined by the rate of diffusion of vapours into wood and the acetylation time rapidly increases with the increase in the wood thickness. Application of gaseous acetylation has therefore remained restricted to thin veneers or solid wood only a few inches long in the fiber direction which can be achieved within a practical treating time.

Traditional acetylation processes that heat pre-impregnated, saturated wood to react the acetic anhydride have poor heating efficiency due to the following problems:

1: The mass of acetic anhydride in the wood.

2: The poor heat conductivity of wood.

3: The fact that the wood heating is by conduction rather than convection.

4: Heat distribution is not uniform throughout the solid wood and thus acetylation is uneven.

5: Over all treatment, reaction and recovery period is extremely long.

It is an object of the present invention to address at least some of these aforementioned shortcomings or to provide the public with a useful choice.

SUMMARY OF THE INVENTION

In broad terms in one aspect the invention provides a process for the impregnation of a working solution into a material including the step of exposing the material to the impregnant at above the atmospheric boiling point of the impregnant at an elevated pressure sufficient to maintain the impregnant in the liquid phase.

In another aspect the invention provides a process for impregnating wood or wood based material comprising the steps of:

- preheating a working solution to a temperature above the atmospheric boiling point ("the super hot temperature") of the working solution, at an elevated pressure sufficient to maintain the impregnant in the liquid phase;
- contacting a wood or wood based material with the working solution at the super hot temperature to cause impregnation of the solution into the wood or wood based material and reaction of the working solution with the wood or wood based material; and

- separating any additional working solution and/or waste material from the wood or wood based material to isolate an impregnated wood or wood based material.

Preferably the process includes the step of, after impregnation, recovering the by-products by releasing the pressure to cause the by-products to boil off. Optionally a vacuum may be applied to improve the recovery of by-products.

Preferably pre-pressure is applied to the wood or wood based material prior to contact with the working solution at the super hot temperature to ensure the working solution remains in the liquid phase.

BRIEF DESCRIPTION OF THE FIGURES

The invention is further described with reference to the accompanying figures in which:

Figure 1 is a schematic illustration of a preferred impregnation process.

Figure 2 is a chart of the boiling pressure at elevated temperature of water, acetic acid and acetic anhydride.

Figure 3 is a photo of a treatment plant of the invention which refers to schematic illustration.

Figure 4 is a close up photo of a treatment plant of the invention.

Figure 5 shows the relationship between acetylation and volumetric Anti Shrink Efficiency (ASE).

Figure 6 shows the relationship between acetylation and increase in oven dried volume.

DETAILED DESCRIPTION OF PREFERRED EMBODIMENTS

In the process of the invention the solution is impregnated into the material in the liquid phase but at above the boiling point of the working solution, at a pressure which will

maintain the working solution in the liquid phase. The solution may be preheated to a temperature above boiling point ("the super hot temperature") under a pressure which ensures the working solution remains in the liquid phase.

The working solution is then released into a pressure impregnation vessel (which has been pre-pressured by a gas, for example nitrogen, to equal or above the pressure of the working solution) containing the wood or wood based material. The elevated temperature will cause rapid reaction of the working solution in the wood while carrying out the impregnation in the liquid phase enables sufficient loading of acetic anhydride in the wood to be achieved to substantially enhance decay and insect resistance and dimensional stability of the wood.

Any additional working solution and/or waste material is separated from the wood or wood based material to protect the working solution from contamination by wood extractives and reaction by-product.

Wood or wood based materials which may be treated by the process of the invention include, but are not limited to solid wood, fiberboard, particle board, wood veneer, wood chips, OSB (oriented strand board), LVL (Laminated Veneer Lumber) and plywood.

Typically the process of the invention will be performed using two or more interconnected pressure vessels. Initially a first vessel contains the working solution and the wood is loaded into a second vessel. Optionally, a third vessel is connected to the second vessel to act as a reservoir for unreacted working solution and waste material.

Preferably the second vessel containing the wood or wood based material is evacuated and pressurized by a non-oxidizing gas, such as nitrogen, prior to contact with the working solution. Suitable gases for applying pressure within the vessels include, but are not limited to, inert gases, for example nitrogen, argon and CO_2. The gas may also be a catalytic gas, for example acetic acid vapour at high temperature or acetic

anhydride itself. The main purpose of introduction of gas or vapours into this vessel is to eliminate boiling off of the super hot solution introduced and consequently stop the working solution cooling. It is believed that the high temperature and the oxidation free environment may induce some additional benefits on both stability and biodeterioration resistance of wood due to thermal treatment.

The composition of the working solution will depend upon the desired effect of the impregnation. Typically, the impregnation process will aim to improve dimensional instability caused by the hygroscopic nature of the wood, discoloration, biological degradation and/or degradation by exposure to UV light. Examples of suitable working solutions include copper naphthenate in solvents above its boiling point such as xylene, isopropanol and methylene chloride. Alternatively, xylene may be used as a heating medium by itself or in combination with paraffin wax. Since high temperature is involved in the process acetic anhydride may also be used to dissolve additional anhydrides, e.g. succinic anhydride. Preferably the working solution is acetic anhydride.

The working solution may be concentrated or alternatively may be diluted in an appropriate solvent, for example isopropyl alcohol (IPA), methylene chloride or water but is not limited thereto.

Desirably, the impregnation of the working solution within the wood or wood based material is assisted by the application of further pressure. The extra pressure can be either hydraulically or pneumatically applied.

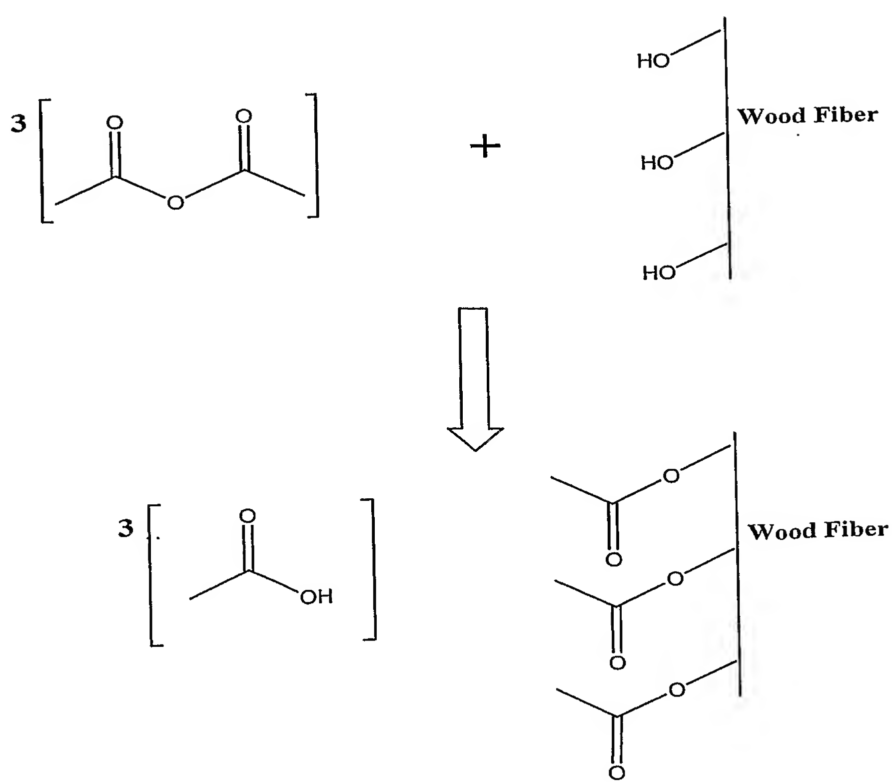
Suitable pressure ranges for the pre-pressure stage are pressures sufficient to stop the solution from boiling at the elevated temperature, which can be from about 10 to about 1000kPa and preferably about 20 to about 700 kPa.

Suitable pressure ranges for the working pressure, i.e. the extra pressure after flooding, are about 20 to about 4000kPa above the initial pre-pressure, but preferably about 20 to about 2000kPa. The working pressure will be partly determined by the permeability of

the wood material being impregnated. Denser woods are less permeable and require higher pressures to achieve satisfactory impregnation of the working solution.

Suitable temperature ranges are about 10°C above the atmospheric pressure boiling point of the working solution and as high as recovery of excess solution warrants. In the case of acetic anhydride the temperature range is from about 150 to about 250°, preferably from about 160 to about 220°C. The most preferred temperature is about 170 to about 200°C.

Preferably, once a desired pressure for maintaining the super hot working solution in a liquid phase has been attained, the pressure and/or temperature are held for a time sufficient to ensure reaction of the working solution within the wood or wood based product. The longer pressure is maintained, the greater the uptake of the working solution by the wood or wood based product. The term 'reaction' refers to any process which locks the working solution or part thereof within the wood or wood based product and may occur by biological, chemical or physical mechanisms. For example, when a working solution of acetic anhydride enters wood, it undergoes substitution reaction below.



The acetyl group of acetic anhydride replaces the hydroxyl group of wood fibers with the formation of acetic acid by-product. The larger size of the acetyl group, compared to the hydroxyl group it replaces, causes a swelling of the wood which improves the properties of the wood, for example by reducing the moisture uptake of the wood.

Desirably any by-products formed by the reaction will have a boiling point lower than that of the working solution. Figure 2 shows that acetic acid (the by-product of the acetylation fixation reaction) has a boiling point lower than that of acetic anhydride. Therefore the pressure of acetic acid will be higher than that of acetic anhydride at any time during holding at high temperature. This facilitates the preferential extraction of by-product from the reaction vessel. It is also believed that the removal of reaction by-product may further encourage the fixation reaction.

Typical waste materials, in addition to reaction by-products, may include naturally occurring wood sugars, hemicelluloses, saccarides and extractives, for example resin and fatty acids.

Desirably the pressure is maintained in the second vessel during the separation and drainage of the working solution and wood materials to prevent kickback from the wood affecting the working solution (the kickback is generally weaker and/or the ingredients are no longer in balance and it may also contain naturally occurring sugars and other molecules present in the wood which are carried back out of the wood). Kickback may occur during the release of pressure ("pressure kickback") or during the application of a vacuum ("vacuum kickback"). By draining the working solution after contact with the wood while maintaining working pressure the extractives and by-product (for example acetic acid) contamination are reduced. Once the working solution has been drained from the second vessel, a vacuum may be drawn through a condenser in a second vessel to facilitate the removal of further unreacted working solution from within the wood or wood based product together with any reaction by-products formed. Where the working solution comprises acetic anhydride vacuum kickback is colourless due to vaporisation condensation of acetic acid rather than liquid flow. This is mainly due to the initial high

temperature of the working solution which facilitates the removals of corrosive unwanted acetic acid.

Extracted reaction by-products may optionally be separated, purified and recycled by methods known in the art. For example, where acetic anhydride is the working solution acetic acid is the by-product. The acetic acid is extracted as a gas and may be collected by condensation. By dehydrating the acetic acid, acetic anhydride is formed suitable for reuse in the impregnation process.

One embodiment of the impregnation process of the invention is shown in Figure 1. Vessel 101 containing working solution (in this example acetic anhydride) is preheated up to about 220°C at a vapour pressure of at least 600 kPa. Vessel 102 containing wood or wood-based material is evacuated and pre-pressurized using nitrogen through 108. Connecting lines 104 and 105 are opened allowing for the pressure between vessels 101 and 102 to equalize as the working solution enters vessel 102.

Once the pressure has equalised and flooding completed, valve 106 is closed and vessel 101 is placed under further pneumatic pressure through valve 109 which, via connecting line 104, in turn further pressurizes vessel 102. The final pressure shall be sufficient to ensure complete penetration of the product being impregnated. Alternatively a pressure pump (111) located in line 104 is used to pressurise cylinder 102. Pressure and temperature are held for a time to cause sufficient reaction of the working solution with the wood based material. Once the reaction is completed, excess working solution is drained from vessel 102 via connecting line 107 to vessel 103. Alternatively the solution can be returned to vessel 101, through line 104, by the pressure pump and/or pneumatic pressure.

Optionally, pneumatic pressure is applied to vessel 102 during draining of the working solution to avoid any kickback. This pressure can be vapour pressure of the super hot solution. Once the working solution has been drained, a vacuum is applied to vessel 102, through condenser 110, to remove any by-product liquid or gas (for example, where acetic anhydride is the working solution, the reaction by-product will be acetic acid. The acetic acid gas removed may be dehydrated to form acetic anhydride for reuse in the process).

In the process described above it is possible to effect treatment (impregnation), bulking (acetic anhydride reaction) and recovery all in one plant. This avoids the need to move the wood between a separate impregnation vessel and heat reaction plant, and also reduces the extra handling of corrosive chemicals.

It is believed that pre-impregnation of air or non-oxidative gas assists the deposition of chemicals where they are necessary (for example at "the cell wall").

When compared to the existing prior art vapour phase acetylation processes, the super hot temperature liquid phase acetylation process of the invention reduces the process time that would otherwise be required at the temperature used to achieve reaction in a single stage or vessel as in a vapour phase impregnation process, while achieving much higher deposit of acetic anhydride in the wood, sufficient to significantly enhance wood resistance to decay and insect attack and dimensional stability for example.

The process can also be used for other preservatives, solvents or combination of the two. For example copper naphthenate can be dissolved in any solvent and pre-heated to above the boiling point of solvent. After flooding and pressurizing using the super hot solvent (above boiling point) the recovery of the solvent is greatly facilitated.

Heat curing at elevated temperature using different solvent or oil and recovery of the solvent using the abovementioned process can bring dimensional stability, water repellency and decay resistance to the wood.

The following description of experimental work further illustrates the invention:

Experiment 1: Acetylation

Radiata pine matched sapwood measuring 300x60x45 mm for ASE (Anti Shrink Efficiency) and 300x20x20 mm for strength test was used in each run for the first 9 treatments. Radiata pine matched sapwood measuring 300x65x45 mm for ASE and two 300x20x20 mm lengths for strength test was used for the treatments 10 to 20.

All wood samples were oven dried prior to treatments except treatments 6, 7 and 20.

Acetylation was conducted in 3 inter connected 2 litre stainless steel cylinders. Cylinders 1 and 2 had a heating jacket connected to PID controller to maintain temperature control. Pneumatic pressures were attained using compressed nitrogen gas.

The volumetric swelling coefficient was calculated according to the following formulae.

$$S = \frac{V_2 - V_1}{V_1} \times 100$$

where S = volumetric swelling coefficient

V2 = wood volume at water saturation

V1 = oven dried wood volume before saturation

Anti Shrink Efficiency was then calculated as follows:

$$ASE = \frac{S_C - S_M}{S_C} \times 100$$

where S_C is volumetric swelling coefficient of control and S_M is volumetric swelling coefficient of modified wood sample.

Details of all treatments are summarized in Table 1

Table 1: Treatment process, weight gain and ASE for experiment 1

Treatment	Solution Temperature Before (C°)	Extra Pressure kPa	Hydraulic Heating	Maximum Temp in Cylinder 2 (C°)	Hydraulic Time (minute)	Pneumatic Time (minute)	Bleeding Time (minute)	Vacuum Time (minute)	Total Treatment Time (minute) Including recovery of by-products	Weight Gain After Treatment (%)	Reacted Weight Gain 48 h oven (%)	Unreacted 48 Hour Oven (%)	Volumetric ASE (%)	Oven Dried Volume Increase (%)	MOE Percentage of Control	MOR Percentage of Control	Burning Inside 0 Min to 5 Max
Con																	0
2	**155**	200	Yes	**162**	30	30	40		**100**	24.6	**19.4**	5.1	**73.5**	7.4	69.1	56.7	3.0
3	**175**	200	Yes	**180**	30	30	40		**100**	28.3	**22.4**	5.9	**87.5**	7.6	75.5	43.1	5.0
4	**180**	200	Yes	**175**	15	25	30		**70**	25.4	**23.2**	2.2	**87.4**	9.6	76.1	56.2	4.0
5	**200**	200	Yes	**159**	20	20	15	25	**80**	32.1	**20.6**	11.6			96.9	83.7	2.0
6*	**170**	200	Yes	**158**	10	20	20	35	**85**	20.9	**7.6**	13.4	**72.1**		76.7	66.7	3.0
7*	**200**	200	Yes	**183**	15	25	10	16	**66**	11.4	**2.9**	8.5	**50.6**		81.2	73.3	4.0
8	Discarded																
9	**190**	500	Yes	**191**	60	1	10	8	**79**	36.7	**29.8**	6.9	**90.9**	12.9	88.0	84.5	3.0
10	**190**	500	Yes	**194**	45	2	10	10	**67**	29.2	**24.9**	4.3	**78.1**	10.7	81.1	76.9	5.0
11	**190**	500	Yes	**170**	10	1	10	20	**41**	25.1	**23.0**	2.1	**75.4**	9.1	93.4	82.5	1.0
12	**180**	500	Yes	**186**	45	1	5	15	**66**	32.8	**28.5**	4.3	**81.1**	10.2	87.2	86.3	1.5
13	**190**	500	No	**130**	5	1	18	15	**39**	22.6	**13.9**	8.7	**62.4**	7.6	105.8	98.9	0.0
14	**200**	500	No	**137**	5	1	16	20	**42**	27.5	**19.6**	7.9	**69.4**	8.3	84.6	71.5	0.5
15	**190**	800	Yes	**159**	10	1	10	20	**41**	26.0	**21.7**	4.3	**74.7**	8.3	71.6	53.2	0.0
16	**200**	500	No	**141**	5	40	5	32	**82**	42.8	**23.4**	19.4	**71.7**	8.3	98.2	92.8	3.0
17	**190**	500	Yes	**161**	10	1	4	28	**43**	22.1	**19.8**	2.3	**69.7**	8.3	95.9	91.1	0.0
18	**190**	500	Yes	**158**	10	1	6	10	**27**	20.6	**18.1**	2.5	**65.7**	7.5	92.6	92.8	0.0
19	**180**	500	Yes	**179**	20	1	5.5	30	**57**	28.2	**25.5**	2.7	**80.4**	9.3	85.1	75.6	1.0
20*	**180**	500	Yes	**178**	20	1	7	30	**58**	10.4	**6.6**	3.7	**64.3**		81.3	74.4	2.0

* Samples were not oven dried prior treatment

The last column of Table 1 in combination with Table 2 illustrate that wood samples with a higher uptake of acetic anhydride exhibit less charring than those samples with a

lower uptake. The rate of reaction was faster than initially anticipated which possibly caused some wood charring in the inner cross-section. By changing treatment parameters such as hydraulic and/or pneumatic temperature, time and pressure, as seen in Table 1, this effect was controlled.

Without wishing to be bound by any particular theory it is believed that the charring of the inner section of wood was due to a combination effect of reaction by-product (acetic acid) and temperature. The temperature component may be further exaggerated by the fact that the acetic anhydride and wood reaction is exothermic, creating acetic acid that is heated even higher than the initial anhydride temperature. In solid wood, the acetic acid by-product trapped inside wood could cause charring.

Treatments with similar schedules but using non-oven dried wood (for example, treatment 19 wood was oven dried and treatment 20 wood was not oven dried with a 8% moisture content) showed more wood charring, possibly due to extra acetic acid production from reaction of acetic anhydride with bound water in the wood.

Two 7mm thin sapwood cross sections were added to treatments 15 and 16. These samples had the same acetylation gain as the larger samples, but no wood charring.

Net uptake, gross uptake, pressure and vacuum kickback of all treatments are summarized in Table 2.

Table 2: Net uptake, Gross uptake, Pressure and Vacuum kickback

Treatment	Net Uptake After Treatment (kg/m3)	Pressure Kickback (kg/m3)	Vacuum Kickback (kg/m3)	Gross Uptake During Treatment (kg/m3)
2	84.87	166.3	120.4	371.6
3	101.01	244.9	35.7	381.6
4	88.12	110.2	32.7	231.0
5	109.39	66.8	72.4	248.7
6		88.8	78.6	
7	44.31	57.1	85.7	187.2
8	Discarded	Discarded	Discarded	Discarded
9	128.99	150.0	140.8	419.8
10	155.38	176.6	125.7	457.7
11	129.51	217.9	54.7	402.1
12	163.78	170.8	103.6	438.3
13	119.27	241.8	84.5	445.6
14	140.89	195.8	91.2	427.8
15	132.38	305.2	118.0	555.6
16	210.58	77.7	99.8	388.1
17	113.40	206.3	92.1	411.9
18	108.94	172.7	77.7	359.4
19	137.61	205.4	102.7	445.7
20	68.46	185.2	91.2	344.9

Treatments that had higher gross uptake showed less wood charring.

Pressure kickback was a red brown colour due to extractives, but vacuum kickback was clear which indicates the vaporization of acetic acid from the wood due to its initial high temperature and its condensation rather than flowing out as liquid (note samples were not pre-extracted to remove extractives).

Figure 3 is an overall plant view of the laboratory scale process of the invention. Work tank 201 contains the working solution for pressurisation and heating prior to contacting the solution with the wood sample within pressure impregnation cylinder 202. By-products recovered from the reaction are removed to cylinder 203 and subsequently

removed from the plant via drainage means 204. A vacuum is drawn on the wood sample through condenser 205.

Figures 5 and 6 show the anti shrink efficiency (ASE) and oven dried volume increase of the wood samples. A limit of 28% weight gain is very close to the maximum theoretical reaction by acetylation. At higher temperatures adopted by this treatment process a weight loss due to solublisation of wood extractives of up to 2% is probable, thus the actual weight gain due to acetylation is probably higher.

Figure 6 shows the close relation of bulking and ASE. Acetylation bulks the wood cell wall and thus the oven dried volume of wood increases. Figure 6 shows this effect, which can be another way of measuring the extent of acetylation. In fact it is believed this volume increase is the main cause of ASE improvement.

Spectra of actual solutions were collected with horizontal ATR cell for treatment 5. Spectra showed that the solution before treatment was 100% acetic anhydride, after treatment solutions showed that the majority of working solution was acetic anhydride, the majority of pressure kickback solution was acetic acid and the vacuum kickback solution was mostly acetic acid (by-product).

Results show that at this preliminary stage a maximum acetylation of radiata pine sapwood can be achieved in a very short time. The fundamentals of this process can be used for preservative treatment and solvent recovery or heat curing of wood by solvents or waxes.

Experiment 2: Alternative Solvents with preservative agent

Two solvents, Isopropyl alcohol and methylene chloride were tried with copper naphthenate as the preservative. One piece of radiata pine sapwood end-sealed 290x72x44mm was treated in each treatment.

A total of five treatments were conducted, 3 Isopropyl alcohol and 2 Methylene Chloride. Treatment time for all treatments was 35 minutes. Cylinder 2 was pre heated to 70°C for all treatments.

Initial pneumatic pre-pressure for all treatments was adjusted to the boiling pressure of respective solvent at elevated temperature.

Table 3 shows the treatment process and solvent recovery for all treatments.

Table 3 Treatment Process, Net Uptake, Net Copper Naphthenate Uptake and Solvent remaining in the Wood

Treatment	Solvent Temperature (°C)/Pressure (kPa)	Hydraulic Pressure (kPa)	Net Uptake (Kg/m^3)	Net Copper Naphthenate Calculated (Kg/m^3)	Analytical Copper (% w/w) in Wood	Net Solvent Uptake Calculated(Kg/m^3)	Copper Penetration
Isopropyl 1	112/205	280	9.8	7	0.03	2.8	Complete
Isopropyl 2	112/190	290	18.4	13	0.08	5.4	Complete
Isopropyl 3	116/220	380	17.9	13	0.07	4.9	Complete
Methylene 1	88/300	440	16.6	12	0.08	4.6	Complete
Methylene 2	86/295	520	12.9	10	0.09	2.9	Complete

Experiment 3: Further Acetylation and Other Species

A further experiment was conducted on Sitka spruce, Scots pine and radiata pine as in Table 4.

Succinic anhydride also reacts to wood hydroxyl group in the same way as acetic anhydride, but creating no acidic by-product. In treatment 39, 10% succinic anhydride was dissolved in acetic anhydride working solution to observe the reduction of wood charring and effect on other wood properties.

Table 4

	Treatment	Wood Moisture Content	Solution Temperature Before (C°)	Extra Pressure (kPa)	Maximum Temp (C°)	Hydraulic Time (minute)	Pneumatic Time (minute)	Bleeding Time (minute)	Vacuum Time (minute)	Total Time (minute) Including Recovery
	Radiata 37	OD	198	500	155	5	1.0	1.5	40	47.5
	Radiata 38	OD	190	500	167	15	1.0	2	40	58
10% SA*	Radiata 39	OD	191	500	169	16	1.0	2	38	57
Used AA**	Radiata 40	OD	193	500	168	15	1.0	2	38	56
Used AA	Radiata 41	9	190	500	168	16	1.0	2	40	59
	Radiata 48	OD	220	500	168	15	1.0	2	36	56
	Radiata 49	OD	220	500	150	5	1.0	2	50	59
	Scots 1	OD	190	800	162	10	1.0	5	33	49
	Spruce 1	OD	193	800	162	10	1.0	3	40	54
	Scots 2	OD	180	500	139	5	1.0	3	46	55
	Spruce 2	OD	182	500	143	5	1.0	3	44	53

* 10% succinic anhydride dissolved in acetic anhydride

** Acetic anhydride working solution was from previous treatments

The weight gain and ASE of third experiments are in Table 5 below.

Table 5

Treatment	Weight gain Aft Treatment (%)	Reacted Weight Aft Oven (%)	Un reacted (%)	Burning Inside 0 Min to 5 Max	ASE (%)
Scots Pine 1	29.10	23.18	5.92	2.00	82.10
Scots Pine 2	25.39	18.95	6.44	0.00	72.40
Spruce 1	26.33	16.21	10.11	2.00	70.80
Spruce 2	17.86	11.03	6.83	0.00	40.40
Radiata 37	19.11	17.01	2.10	0.50	63.56
Radiata 38	21.60	19.30	2.30	0.25	70.95
Radiata 39	25.10	22.80	2.30	0.00	70.10
Radiata 40	21.00	18.50	2.50	0.00	68.51
Radiata 41	5.50	8.10	5.40	0.25	39.34
Radiata 48	20.4	18.4	2.00	0.00	64.4
Radiata 49	16.95	15.58	1.37	0.00	39.5

531217

Experiment 4: Further Heat Curing by Super Hot Solvent

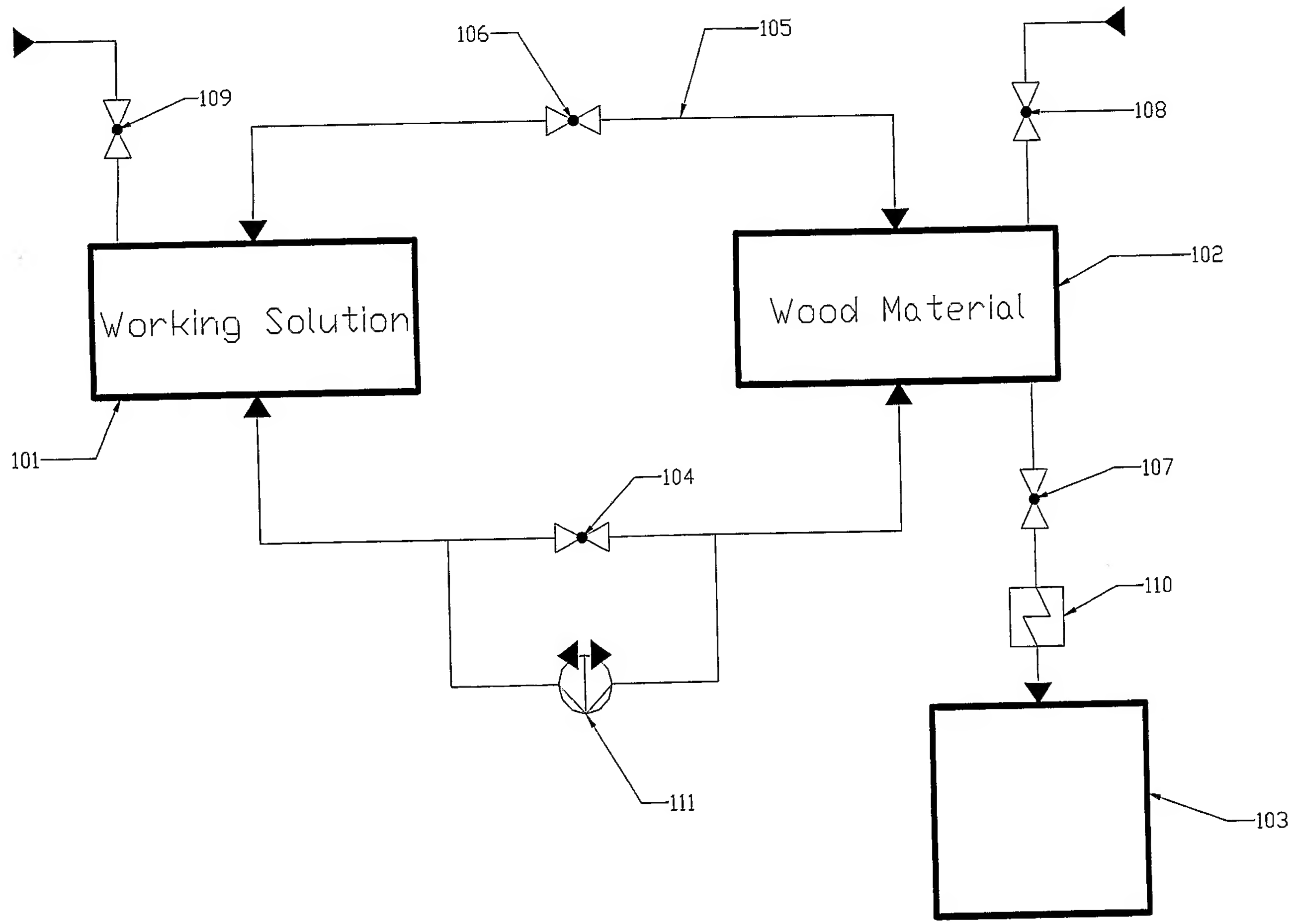
Super heated Xylene at 200°C was used as a medium for heat treatment. Two treatments were conducted, xylene and 5% paraffin wax dissolved in xylene. In both treatments radiata pine sapwood at 9% moisture content was used. Wood was light brown in colour after treatments and lost weight due to extractives dissolved in xylene. Treatment process is summarised in Table 6

Table 6

	Wood Moisture Content	Solution Temperature Before	Extra Pressure	Maximum Temp	Hydraulic Time	Pneumatic Time	Bleeding Time	Vacuum Time	Total Time
xylene	9	201	500	201	30	3.0	10	25	68
xylene+wax	9	202	500	210	30	3.0	8	30	71

ASE for xylene treated sample was 13.2% and for 5% wax dissolved in xylene was 7.8%.

The foregoing describes the invention and a preferred form thereof. Alterations and modifications as will be obvious to those skilled in the art as intended to be incorporated within the scope hereof.



Working Solution
Wood Material
101
102
103
104
105
106
107
108
109
110
111

FIGURE 1

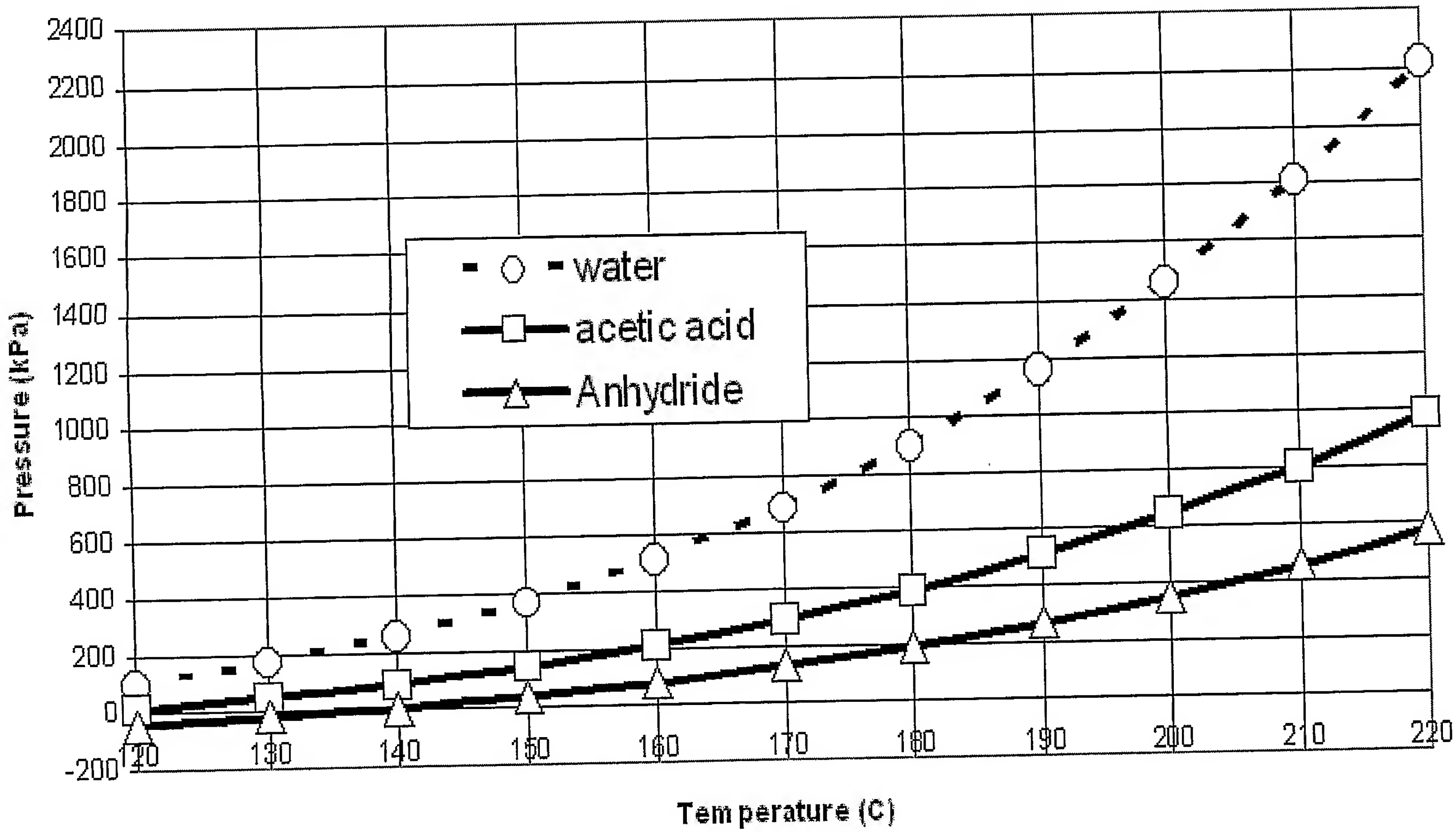

water
acetic acid
Anhydride
Pressure (kPa)
Temperature (C)
2400
2200
2000
1800
1600
1400
1200
1000
800
600
400
200
0
-200
120
130
140
150
160
170
180
190
200
210
220

FIGURE 2

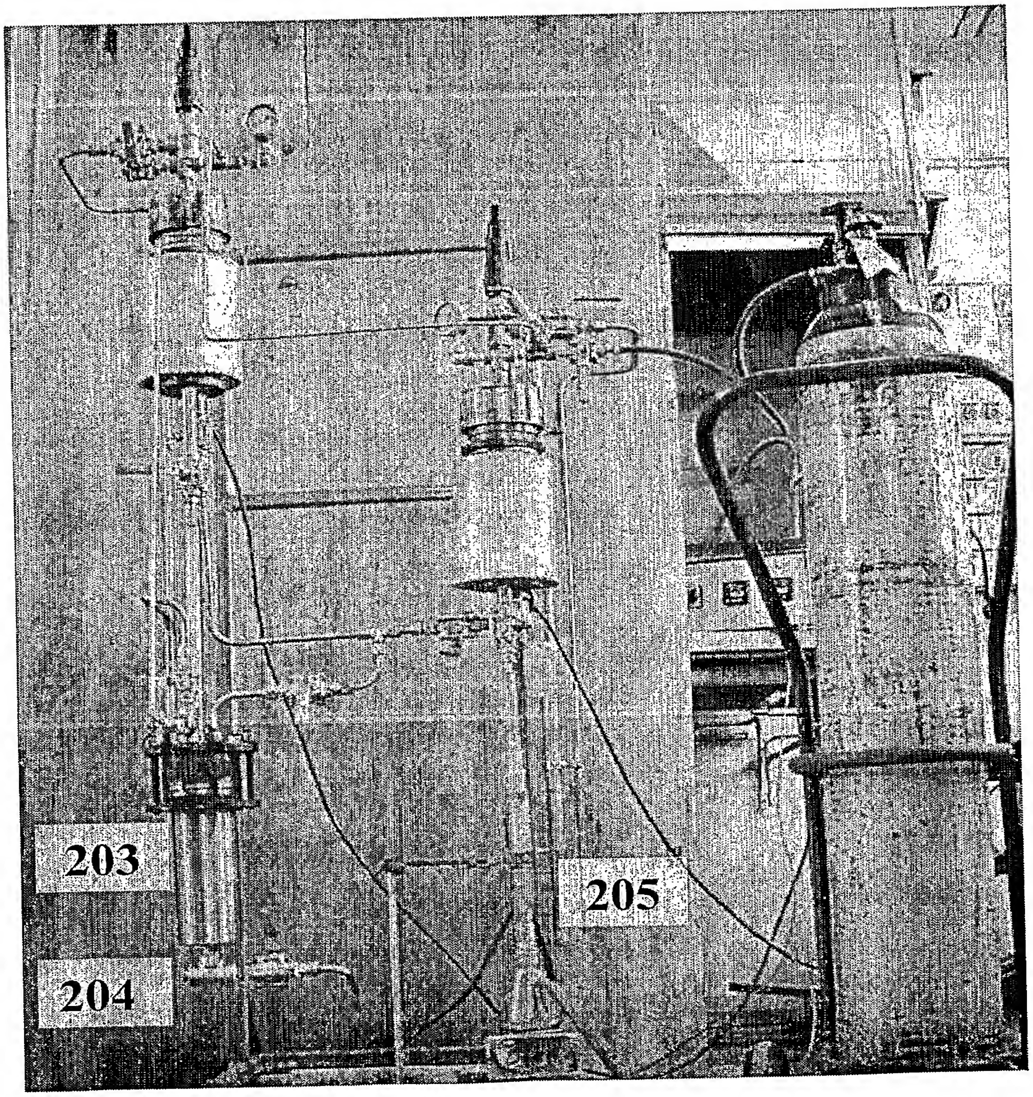


FIGURE 3

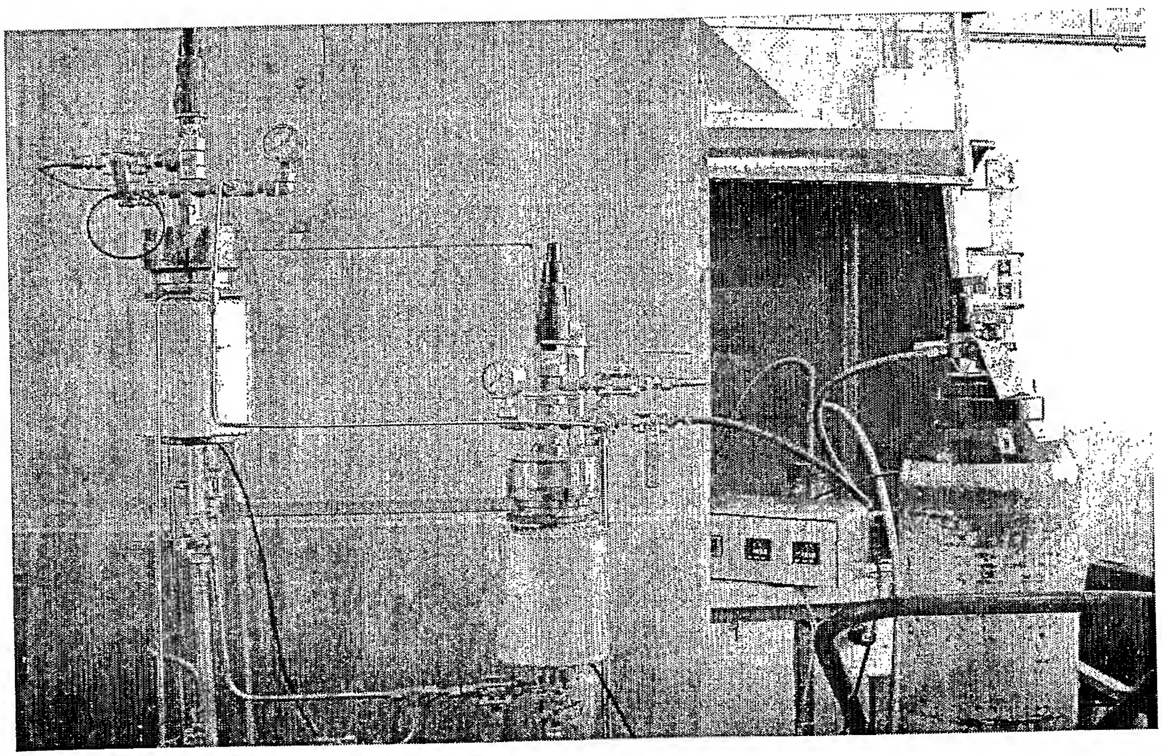


FIGURE 4

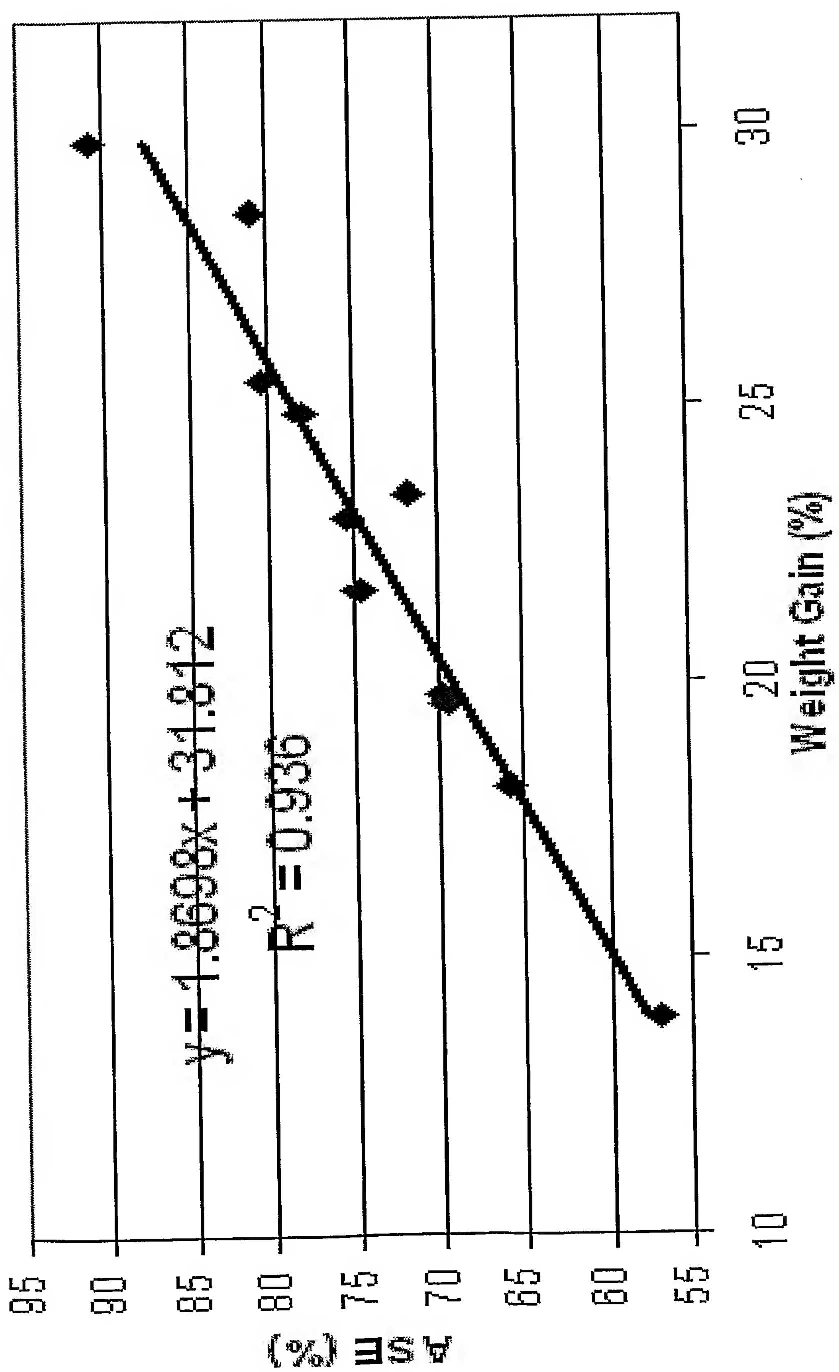

y = 1.8608x + 31.812
R^2 = 0.936
A S E (%)
95
90
85
80
75
70
65
60
55
10
15
20
25
30
Weight Gain (%)

FIGURE 5

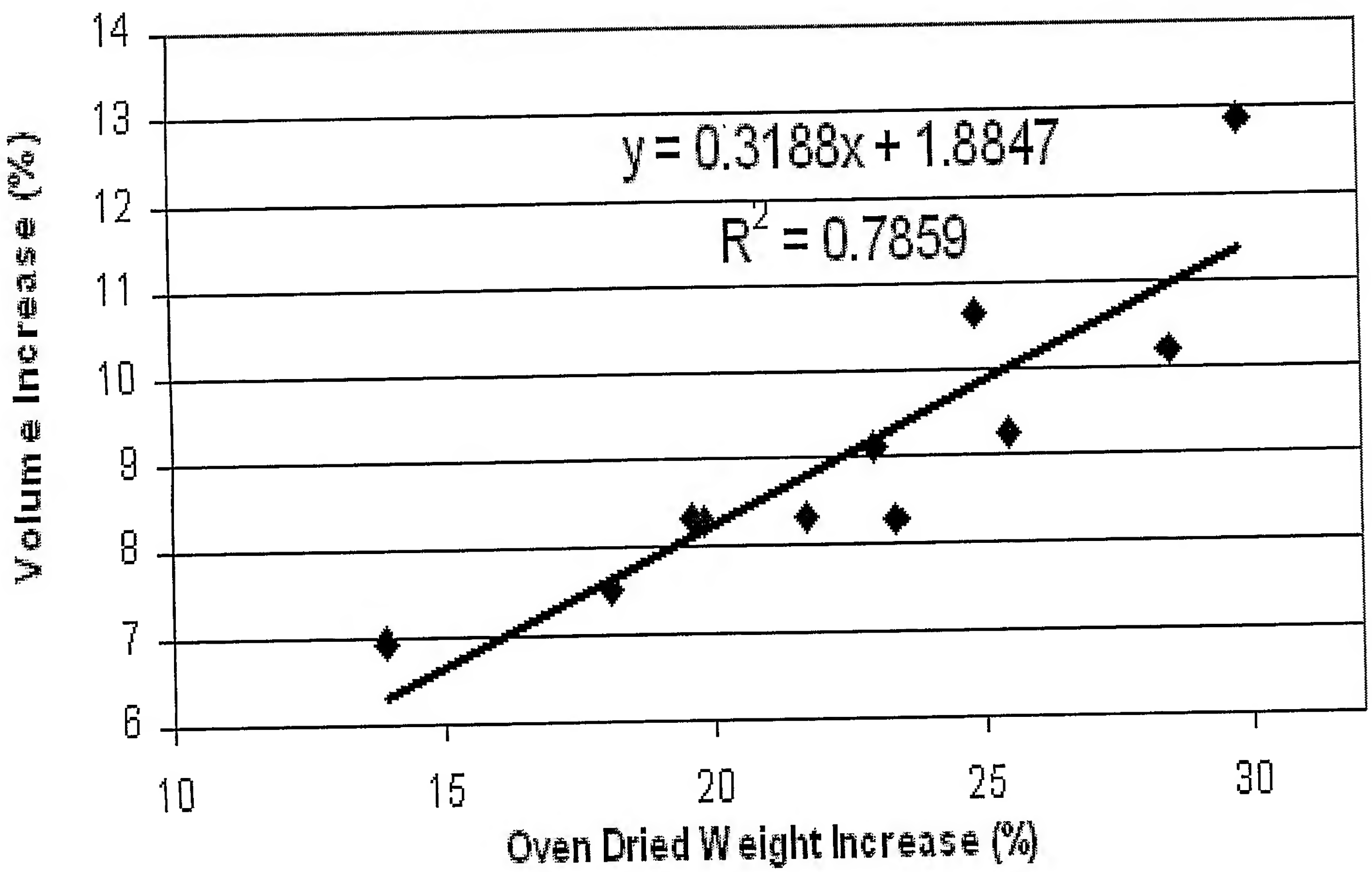

y = 0.3188x + 1.8847
R2 = 0.7859
Volume Increase (%)
14
13
12
11
10
9
8
7
6
10
15
20
25
30
Oven Dried Weight Increase (%)

FIGURE 6